# Recent Advances in High-*k* Nanocomposite Materials for Embedded Capacitor Applications

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## ABSTRACT

**In this paper, a wide variety of high dielectric constant (*k*) composite materials which have been developed and evaluated for embedded capacitor application are reviewed. Current research efforts toward achieving high dielectric performance including high-*k* and low dielectric loss for polymer composites are presented. New insights into the effect of unique properties of the nanoparticle filler, filler modification and the dispersion between filler and polymer matrix on the dielectric properties of the nanocomposites are discussed in details.**

Index Terms — **Dielectric materials, capacitors, nanocomposites, nanotechnology.**

## 1 INTRODUCTION

**HIGH** dielectric constant (*k*) materials have received increasing interest recently for various potential applications including gate dielectrics, high charge-storage capacitor and electroactive materials [1-3]. For instance, materials with high dielectric constant and low dielectric loss are imperative for real applications of embedded passives, specifically capacitor, which is one of the emerging and important technology for microelectronic system integration to provide the size reduction and performance enhancement advantage of many electronic systems [4,5].

Driven by ever growing demands of miniaturization, increased functionality, high performance and low cost for microelectronic products and packaging, new and unique solutions in system integration, such as system-on-chip (SOC) and system-in-package (SiP), have been hot topics. Despite the high level of integration, the number of discrete passive components (resistors, capacitors or inductors) remains very high. Embedded passives, an alternative to discrete passives, can address issues associated with discrete parts, including substrate board space, cost, handling, system performance, assembly time and yield. Figure 1 schematically shows an example of realization of embedded passive technology by integrating resistor and capacitor films into the laminate substrates [4,5].

By removing these discrete passive components from the substrate surface and embedding them into the inner layers of substrate board, embedded passives can not only provide the advantage of size and weight reduction, but also have many other benefits such as increased reliability, improved performance and reduced cost, which have driven a significant amount of effort during the past decade for this technology.

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However, embedded passive technology has not been commercialized for electronic packages yet due to materials and

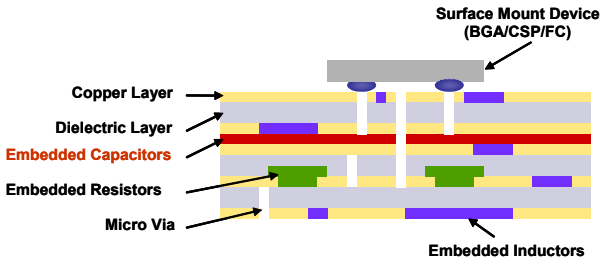


**Figure 1.** Schematic illustration of embedded passives integrated into the laminate substrate.

commercialized for electronic packages yet due to materials and process issues. Therefore, it is necessary to develop materials that satisfy the requirements of fabrication as well as electrical and mechanical performances to enable embedded passive technology [2,6]. Requirements for real applications of embedded capacitors include high *k* low dielectric loss, low leakage current, high breakdown voltage and sufficient stability. Materials challenges include dielectrics with *k* above 1000 [6], and low dielectric loss. Generally, a dissipation factor under 0.1% is considered to be quite low and 5% is high [4]. Very low dissipation factor is desired for RF applications to avoid signal losses, but much higher values can be tolerated for decoupling applications.

In this paper, a wide variety of high-*k* composite materials which have been developed and evaluated for embedded capacitor application are reviewed and discussed. Current research efforts toward achieving polymer composites which fulfill the balance between sufficiently high *k* and low dielectric loss are presented.

# 2 HIGH-*k* MATERIALS FOR EMBEDDED CAPACITORS

From a dielectric property point of view, dielectric constant and dielectric loss are the two most important parameters that dictate the performance of a candidate material for embedded capacitors. To meet the stringent materials requirements, considerable attention has been devoted to the research and development of the candidate high-*k* materials. A wide variety of materials have been studied and evaluated for this application.

In the beginning, ferroelectric ceramic materials with permanent dipole moment which gives these materials high-*k* in the thousands, including $BaTiO_3$ (barium titanate), $BaSrTiO_3$ (barium strontium titanate), $PbZrTiO_3$ (lead zirconate titanate) etc., have been used as dielectric materials for decoupling capacitors [4, 8]. However, very high processing temperature (>600 °C) required for sintering is unsuitable for the embedded capacitor applications in the low cost organic PCB industry.

Polymer dielectric materials are compatible with PCB manufacturing, but the *k* value of general polymers is too low (<5) to achieve high capacitance density. To raise the *k* of polymer-based materials, polymer composite materials provide an ideal solution. The methodology of this approach is to combine the advantages from the polymers which meet the requirements for the low cost organic substrate process, i.e. low temperature processibility, mechanical flexibility and low cost, with the advantages from the fillers, such as desirable dielectric and electrical properties.

## 2.2 FERROELECTRIC CERAMIC/POLYMER COMPOSITES

Ferroelectric ceramic/polymer composites that adopt traditional ceramics as fillers, e.g. $BaTiO_3$, $BaSrTiO_3$, $PbZrTiO_3$, have been actively explored as a major material candidate [2,7,9-15]. Some challenging issues in these polymer composites for high *k* applications still exist, such as limited dielectric constants, low adhesion strength and poor processibility. Most of the *k* values of polymer-ceramic composites developed to date are below 100 at room temperature. By employing the relatively high *k* polymer matrix, the *k* values of ceramic/polymer composites can be effectively enhanced because the *k* of polymer matrix shows very strong influence on the *k* of the final composites [2,12]. For instance, poly(vinylidene fluoride-trifluoroethylene) (P(VDF-TrFE)) copolymer, a class of relaxor ferroelectric, can have a relatively high room temperature *k* (~40) after irradiation treatment [16]. Bai et al. prepared $Pb(Mg_{1/3}Nb_{2/3})O_3$-$PbTiO_3$/P(VDF-TrFE) composites with *k* values above 200 at 10 kHz [2]. Rao et al. reported a lead magnesium niobate-lead titanate (PMN-PT, 900 nm)+$BaTiO_3$ (50 nm)/high-*k* epoxy system (effective *k*: 6.4) composite with *k* value about 150 at 10 kHz, in which ceramic filler loading as high as 85% by volume [17]. A major concern for ceramic/polymer composites lies in that the high filler loading of ceramic powders will lead to some technical barriers for real application in the organic substrate, including poor dispersion of the filler within the organic matrix, and poor adhesion of the dielectric layer to other layers in PCB.

## 2.3 CONDUCTIVE FILLER/POLYMER COMPOSITES

Conductive filler/polymer composite is another approach towards high-*k* materials for embedded capacitor application, which is a kind of conductor-insulator composite based on percolation theory [18]. Ultra-high *k* can be expected with conductive filler/polymer composites when the concentration of the conductive filler is approaching the percolation threshold. Sometimes the effective *k* of the metal-insulator composite could be three or four magnitudes higher than the *k* of the insulating polymer matrix. And also this percolative approach requires much lower volume concentration of the filler compared to traditional approach of high-*k* particles in a polymer matrix. Therefore, this material option represents advantageous characteristics over the conventional ceramic/polymer composites, specifically, ultra-high *k* with balanced mechanical properties including the adhesion strength. Various conductive fillers, such as silver (Ag), aluminum (Al), nickel (Ni), carbon black, have been used to prepare the polymer-conductive filler composites or three-phase percolative composite systems [18, 20-25]. For instance, Dang et al. [23] and Rao et al. [18] reported *k* value of 400 and 2000 in Ni/PVDF composite and Ag flake/epoxy composite, respectively. Although these composites were reported with high *k* values, they still cannot be considered as effective materials for embedded capacitor applications due to the accompanied high dielectric loss tangent and conductivity. Currently, much work has been focused to develop dielectric materials which fulfill the balance between sufficiently high *k* and low dielectric loss, and satisfy the requirements to be a feasible option for embedded capacitor applications. The approaches to control the dielectric loss will be discussed in details in session 3.2.

## 2.4 All-ORGANIC POLYMER COMPOSITES

The composites fabricated by dispersing an organic filler material possessing very high dielectric constant in a polymer matrix can exhibit high-*k* as well. Zhang et al. selected copper-phthalocyanine (CuPc) oligomer, a class of organic semiconductor materials with *k* as high as $10^5$, as high-*k* filler and dispersed in P(VDF-TrFE) matrix. The composite showed a *k* of 225 and a loss factor of 0.4 at 1 Hz [3]. The high dielectric loss is due to the long-range intermolecular hopping of electron. Wang et al. further chemically modified CuPc and bonded to P(VDF-TrFE) backbone to improve the dispersion of CuPc in polymer matrix. Dielectric loss was reduced and dielectric dispersion over frequency was weakened for chemically modified CuPc/P(VDF-TrFE) composites [26]. A *k* value above 1000 (at 1 kHz) has been achieved by Huang et al in an all-polymer high-*k* percolative composite material, fabricated by a combination of conductive polyaniline (PANI) with a poly (vinylidene fluoride-trifluoroethylene-chlorotrifluoroethylene) [P(VDF-TrFE-CTFE)] terpolymer matrix ($k$ > 50) [27]. Lu et al. reported a PANI/epoxy composite prepared by *in-situ* polymerization with a high dielectric constant close to 3000, a dielectric loss tangent less than 0.5 at room temperature and 10 kHz [28]. The possibility of all-organic composites as candidate high-*k* material for embedded capacitor requires further investigation and demonstration.

**Table 1.** Composition and dielectric properties of high-*k* nanocomposite materials.

| Materials | *K* | Tan $\delta$ | Filler Size | Filler Loading | Ref. |
|---|---|---|---|---|---|
| $BaTiO_3$/epoxy | 40 (1 Hz) | 0.035 (1 Hz) | 100-200 nm | 60 vol% | 11 |
| $Pb(Mg_{1/3}Nb_{2/3})O_3$-$PbTiO_3$/P(VDF-TrFE) | ~200 (10 kHz) | 0.1 (10 kHz) | 0.5 $\mu$m | 50 vol% | 2 |
| bimodal $BaTiO_3$/epoxy | 90 (100 kHz) | 0.03 (100 kHz) | 916 nm+60 | 75 vol% | 16 |
| PMN-PT+$BaTiO_3$/ high-*k* epoxy | ~150 (10 kHz) | N/A | 900nm/50 nm | 85 vol% | 17 |
| $BaTiO_3$/ P(VDF-HFP) | 37 (1 kHz) | < 0.07 (1 MHz) | 30-50 nm | 50 vol% | 31 |
| Carbon black/epoxy | 13000 (10 kHz) | 3.5 (10 kHz) | ~30 nm | 15 vol% | 25 |
| Ag/carbon black (CB)/epoxy | 2260 (10 kHz) | 0.45 (10 kHz) | Ag: 13 nm | Ag: 3.7 wt%, CB: 20 wt% | 21 |
| Al/Ag-epoxy | 160 (10 kHz) | 0.045 (10 kHz) | Ag: <20 nm | Al: 80 wt% | 33,34 |
| Ag/epoxy | ~300 (1 kHz) | 0.05 (1 kHz) | 40 nm | 22 vol% | 20 |
| Ag@C/epoxy | >300 (1 kHz) | < 0.05 (1 kHz) | 80-90 nm core | 25-30 vol% | 32 |

# 3 RECENT ADVANCES IN HIGH-K NANOMATERIALS

## 3.1. NANOPARTICLE-BASED HI-*k* COMPOSITE MATERIALS

During recent years, great efforts have been made towards the synthesis and application of nanoparticles because of their unusual physical and chemical properties. Polymer composite materials based on nanoparticles provide a potential solution to meet the present and future technological demand in terms of the good processibility and mechanical properties of polymers combined with the unique electrical, magnetic or dielectric properties of nanoparticles [29]. Additionally, nano-sized particles are preferred for high-*k* dielectric composite materials because they could help achieve thinner dielectric films leading to a higher capacitance density. Therefore, more nanoparticles of ceramic, metallic or even organic semiconductor have been introduced to prepare high-*k* dielectric materials recently. It is anticipated that as-prepared nanocomposites are highly promising nanodielectrics, which is a new class of dielectric material [30]. Some of the examples are summarized in Table 1.

In the past decade, a great deal of effort has been devoted to the development of ceramic/polymer (0-3 connectivity) composites, but most of the ceramic fillers used are in the micron size range. Although finer particle size is required to obtain a thin dielectric film and to increase the capacitance density, extremely fine ceramic particles may lead to the change of crystal structure from tetragonal, which results in the high permittivity, to cubic or pseudo-cubic. Generally speaking, the tetragonality and hence the permittivity of ceramic particles decreases with the particle size. Cho et al. prepared $BaTiO_3$/epoxy composite embedded capacitor films (ECFs) with average particle size of 916 nm and 60 nm, the *k* values of ECFs made of 916nm particles were higher than those made of 60 nm particles. So the coarser particle is more useful than the finer particle to obtain high *k* of ECFs using unimodal powder. But by adopting bimodal fillers, fine nanopariticle can effectively enhance the *k* values by maximizing packing density and removing the voids and pores formed in the dielectric films. A dielectric constant of about 90 was obtained at a frequency of 100 kHz using these two different size $BaTiO_3$ powders [15].

Conductive filler/polymer nanocomposites have been identified as a promising method to fulfill the material requirements for embedded capacitors. However, the dielectric loss of this type of materials is very difficult to control, because the highly conductive particles are easy to form a conductive path in the composite as the filler concentration approaches the percolation threshold. Therefore, high dielectric loss and narrow processing window of this system have plagued the metal/polymer composites from real applications. To solve the problems of the conductive filler/polymer composites, currently much work has been directed to the control of the dielectric loss of this system to overcome the above-mentioned drawbacks. A wide variety approaches to achieve high dielectric performances including high-*k* and low dielectric loss for these material systems are discussed in the following section 3.2.

## 3.2. PERFORMANCE ENHANCEMENT OF HIH-K NANOCOMPOSITES

### 3.2.1. CONTROLLED DISPERSION

Uniform dispersion of nanoparticles in nanocomposite materials is required because nanoparticle agglomerates will lead to undesirable electrical or materials properties. Therefore, dispersion of nanoparticles is an extremely important contributor for achieving improved dielectric properties and reproducibility. Addition of surfactant or dispersant such as phosphate esters can improve the dispersion of nanoparticles in polymer matrix and thereby the overall film quality and dielectric performance of the nanocompsites [35].

Chemical modification of nanoparticles is a useful approach to facilitate the dispersion of nanoparticles as well. For instance, Kim et al. reported that surface modification of $BaTiO_3$ and related perovskite-type metal oxide nanoparticles with phosphonic acid ligands leads to well-dispersed $BaTiO_3$/polymer nanocomposite films with high dielectric strength [31]. This methodology is straightforward and

easily adapted to a wide range of systems by choosing appropriate ligand functionality. Another example is related with CuPc as discussed in 2.4. Chemically modified CuPc can improve the dispersion of CuPc in polymer matrix. Compared to the simple blending method, the CuPc oligomer particulates in grafted sample are of relatively uniformly size in the range of 60-120 nm, which is about 5 times smaller than that of blended composite. Furthermore, dielectric loss was reduced and dielectric dispersion over frequency was weakened [26].

### 3.2.2 CORE-SHELL HYBRID FILLER

The direct contact of the conductive metal fillers will lead to high dielectric loss or even conduction for the conductive filler/polymer composites at or above percolation threshold. Therefore, core-shell structured filler was proposed and studied to be utilized as fillers instead of using conductive filler directly because the non-conductive shell could serve as electrical barriers between the conductive cores to form a continuous interparticle-barrier-layer network and thus achieve high-*k* and low loss. The core/shell structure can be formed either pristine or by synthesis. Xu et al. developed high-*k* polymer composite materials using self-passivation Al as the filler. The self-passivated insulating aluminum oxide layer on the Al metallic core showed significant effects on dielectric properties of the corresponding composites. For composite containing 80 wt.% Al, a *k* of 109 and a low dielectric loss tangent of about 0.02 (@10 kHz) were achieved [22]. Most recently, Shen et al. reported a new polymer composite using synthesized core/shell hybrid particles, metal Ag cores coated by organic dielectric shells, as fillers. The organic dielectric shells act as interparticle barriers to prevent the direct connection of Ag particles and facilitate the dispersion of fillers in the polymer matrix as well, leading to stable high-*k* (>300) and rather low dielectric loss tangent (<0.05) for the polymer dielectric [32].

### 3.2.3. SURFACE-MODIFIED METAL FILLER

Similarly, attempts have been made to reduce the dielectric loss of the conductive filler/polymer nanocomposites by introducing surfactant layer coated on the metal filler surfaces during nanoparticle synthesis. For example, Qi et al. reported a Ag/epoxy nanocomposite with 22 vol.% of Ag possessing a *k* of 308 and a relatively low dielectric loss of 0.05 at a frequency of 1 kHz [20]. In this material system, 40 nm Ag nanoparticles coated with a thin layer of mercaptosuccinic acid were randomly distributed in the polymer matrix. The *k* and dielectric loss increase with the filler concentration up to 22 vol.%. The decrease of *k* after that point is not due to conduction, and this is attributed to the porosity as revealed from miscrostructure investigation. The introduction of porosity is possibly caused by the absorbed surfactant layer which leaves space between Ag particles and voids which are not occupied by polymer. Another contributor could be micropores formed from solvent residue during curing, especially at a higher Ag content. In addition, no rapid increase of the dielectric loss tangent values was observed. Therefore, the observed highest *k* value was not considered as a real percolation threshold and the formation of a conducting filler network was prevented by the surfactant coating layer.

### 3.2.4. EFFECT OF METAL NANOPARTICLES

Incorporation of metal nanoparticles in the high-k materials might bring out some interesting phenomenon due to the unique properties of the nanoparticles. Lu et al. reported that incorporation of ultra-fine sized Ag nanoparticles in the Ag/carbon black/epoxy nanocomposites increased the *k* value and decreased the dielectric loss tangent as shown in Figure 2 [21]. The sample with 3.7 wt.% Ag showed *k* of 2260 while the Df (dissipation factor) was maintained at around 0.45, which is much lower than the control sample without Ag nanoparticles (*k*: 1600, Df: 0.7). The remarkably increased *k* of the nanocomposites was due to the piling of charges at the extended interface of the interfacial polarization-based composites. The reduced dielectric loss might be due to the Coulomb blockade effect of the containing Ag nanoparticles, a well-known quantum effect of metal nanoparticles [36,37].

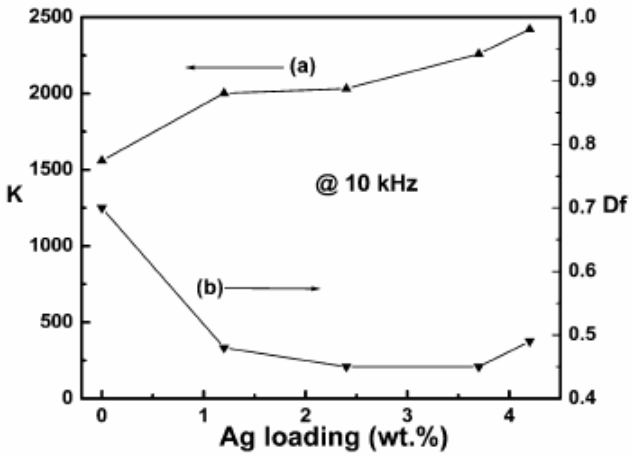


**Figure 2.** Variation of *k* and Df at 10 kHz with different loading level of Ag nanoparticles [21].

In addition, the presence of the capping agent and its ratio with respect to the metal precursor were found to have great effect on the size, and their distributions of the synthesized Ag nanoparticles in the nanocomposites (see Figure 3). In the case of R = 1 and 0.6 (see Figures 3a and 3b), Ag nanoparticles of 1-3 nm were well-dispersed, and the agglomeration of the small particles formed larger clusters of the size ranging 10-15 nm. On the other hand, both small and large particles in size of 1-3 nm and 35-45 nm, respectively, were observed in the same samples with smaller Rs (see Figures 3c and 3d). For Ag/CB/epoxy composites containing 4.2 wt.% Ag and 19.6 wt. % carbon black, smaller size and narrower size distribution of Ag nanoparticles resulted in more reduced dielectric loss as displayed in Figure 4 [21].

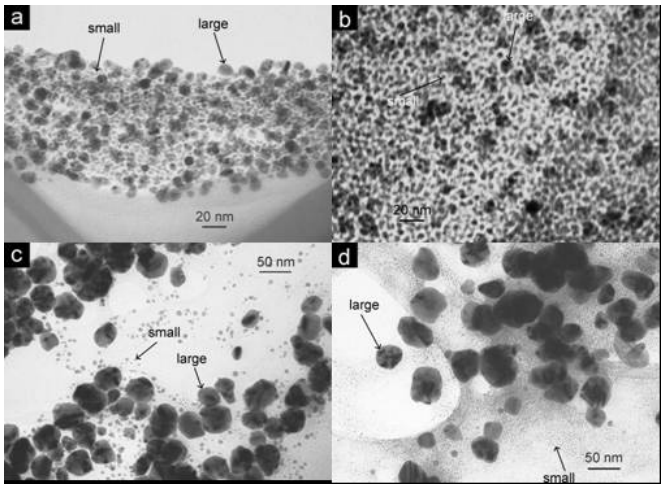


**Figure 3.** Ttransmission electron microscopy (TEM) micrographs of Ag nanoparticles in the epoxy matrix in the presence of a capping agent with [capping agent]/[Ag precusor] ratio (a) R = 1, (b) R = 0.6, (c) R = 0.4 and (d) R = 0.2 [21].

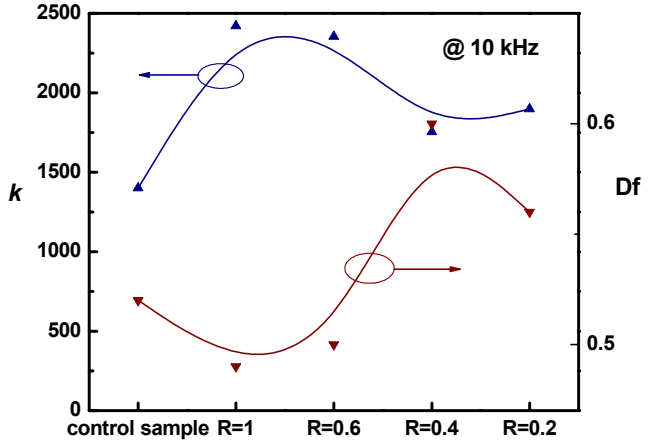


**Figure 4.** *k* and Df values of Ag/CB/epoxy composites with various concentrations of a capping agent (R= [CA]/[$AgNO_3$]).

### 3.2.5. HIGH-*k* POLYMER MATRIX

As mentioned in previous section, the *k* values of ceramic/polymer composites can be effectively enhanced by employing the relatively high *k* polymer matrix. The high-*k* polymer matrix can be either intrinsic [2] or extrinsic by incorporation of additional reagent/filler in the polymer [17]. Similarly, the effective *k* of the conductive filler-polymer composite can be further enhanced by increasing the *k* value of the polymer matrix. Lu et al prepared polymer nanocomposite as high-*k* polymer matrix, which is composed of polymer matrix and an appropriate amount of conductive filler [33,34]. Ag nanoparticles were generated by *in-situ* photochemical reduction of a metallic precursor within the polymer matrix. Figure 5 displays TEM micrographs of Ag nanoparticles synthesized via this method in an epoxy resin. Nanoparticle size ranged from 15 to 20 nm with smaller particles down to 3-5 nm. The composition of *in-situ* formed nanoparticles in the whole mixture was estimated at around 10 wt%.

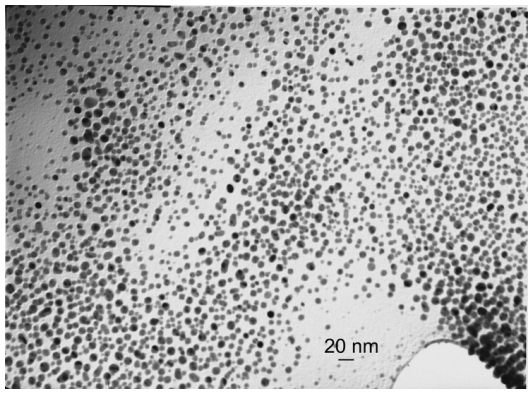


**Figure 5.** TEM micrographs of Ag nanoparticles synthesized within epoxy resin via in-situ photochemical reduction method [33].

Self-passivated Al particles were then incorporated in the as-prepared Ag-epoxy nanocomposite to further improve the *k* of Al/epoxy composites while maintaining the relatively low dielectric loss. Figure 6 displays the dielectric properties of Al/epoxy and Al/Ag-epoxy composites with different Al filler loading at a frequency of 10 kHz. The composites showed a more than 50 % increase in *k* values compared with an Al/epoxy composite with the same filler loading of Al. Moreover, the dielectric loss was maintained below 0.1. The results suggested that the *in-situ* formed Ag-polymer nanocomposites can be employed as a high-*k* polymer matrix to host various fillers such as conductive metal or ferroelectric ceramic fillers to achieve both high *k* and relatively low dielectric loss.

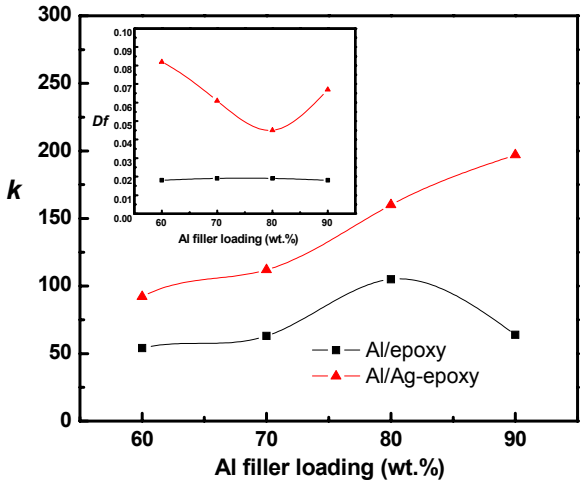


**Figure 6.** *k* and Df (see the inset) values of Al/epoxy and Al/Ag-epoxy composites with different Al filler loading (@ 10 kHz) [34].

## 4 SUMMARY

This paper reviewed recent advances and trend in the field of high-*k* materials for the embedded capacitor applications. Generally speaking, high-*k* materials which meet the requirements for this application should possess high dielectric constant, low dissipation factor, high thermal stability, simple processibility, and good dielectric properties over broad frequency range. However, no such ideal materials that satisfy the above-mentioned prerequisites simultaneously have been realized till present. Polymer composite materials have been studied extensively. Efforts to improve the overall

dielectric performance of these candidate materials have been devoted to maximize the dielectric constant and suppress the dielectric loss. New insights into the unique properties of the nanoparticle filler, filler modification and the dispersion between filler and polymer matrix have been gained in most recent studies. Specifically, these techniques include: (1) optimized formulation of dielectric materials with high filler loading of high dielectric constant ceramics for ceramic-polymer nanocomposites and appropriate loading level of conductive fillers in the neighborhood of percolation threshold for conductive filler-polymer nanocomposites; (2) improvement in microstructure of dielectric materials including filler size and distribution, packing, and dispersion in the polymer matrix; (3) enhancement of *k* values of nanocomposites by employing high-*k* polymer matrix; (4) modification of the filler interface to facilitate dispersion in the polymer matrix and suppress the dielectric loss of the composite materials.

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